

Maxwell, V., Ellam, R.M. , Skarpelis, N. and Sampson, A. (2019) The context and nature of the evidence for metalworking from mid 4th Millennium Yali (Nissyros). *Journal of Greek Archaeology*, 4, pp. 1-30. (doi: 10.32028/9781789693775-2)

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Deposited on 18 June 2020

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The Context and Nature of the Evidence for Metalworking from Mid 4th Millennium Yali (Nissyros)

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ABSTRACT

Two crucibles with copper adhering (and one lead rivet) have been found on Yali (Nissyros) dating to the Final Neolithic, mid-4th millennium BCE. This is important and rare evidence for the earliest phase of Aegean metallurgy, now recognized as emerging in circumstances of high mobility and variable technological preference and practice. The finds are presented here through a study of their context, typology and chemical and lead isotope analysis. The results show that the crucibles come from the main settlement on the island; they were locally made, using a clay recipe deliberately tailored to the needs of metalworking. The copper was pure, with low levels of naturally occurring arsenic. The copper and lead came from the same source which, on current evidence, appears to be to Kythnos. The community on Yali was in contact with a broader Aegean where multiple metallurgical technologies are known. The presence of tin ore, or its product, might be indicated. Though small in scale, there are some parallels with the nature and technology of metallurgical activities in the succeeding Early Bronze Age.

Introduction

In the wider Aegean, it is now recognized that the very end of the Neolithic is a key period in the evolution of communities and in the roots of changes observed in the succeeding Early Bronze Age. One important aspect of this change was involvement in metallurgy. Establishing the nature of early metallurgy could enrich our understanding of the processes of change at work at this time.

This article presents the study of two ceramic crucibles, with copper adhering, and one lead rivet from a mid-4th millennium BC context in the Aegean. The aim is to better understand their place in the emergence of metallurgy in the region through a study of context, typology and chemical and lead isotope analysis.

The crucibles and lead were found in the 1980's, during rescue excavations prompted by the extensive industrial quarrying of the island of Yali (off Nissyros) in the east Aegean,¹ FIGURE 1. Evidence for occupation of Yali dates from c.4500-c.3200 generally; the evidence for metalworking dates to c.3500 BC, specifically. They were found in a settlement with an occupation phase of approximately three hundred years.²

Context

The chronological and theoretical context of the evidence for metalwork within the broad Aegean is first briefly reviewed, followed by the spatial, depositional and use context of the crucibles.

Concerning the chronological context, Muhly noted how, 'the past decade has witnessed a transformation in our understanding of the chronology of the Neolithic period in Greece and Anatolia and....this has had a profound impact on our evaluation of the development of metallurgical technology in both areas.'³ The chronology of the long Final Neolithic (FN) (c.4500-3200BC) is still evolving. This has given rise to a range of chronological schemes, each with their own nomenclature. However, there is some lack of consensus amongst researchers as to the nature and timing of development within the region, FIGURE 2.

¹ Sampson 1988, 127, plate 38; 187, 3 and 188, 4

² Sampson 2008

³ Muhly 2006, 160

The chronological system devised by the excavator,⁴ and used for Yali, is one example of such schemata. It initially named the evidence for the final phases of the Neolithic as Late Aegean Neolithic (LAN) with later refinement of this singular chronology to Late Neolithic II a/ b.⁵

To tackle the chronological disharmony between Crete and other Aegean regions, Tomkins⁶ abandoned the traditional chronological terminology there in favour of a system compatible with that used elsewhere in Greece and the Aegean. Greater chronological resolution has been achieved for the FN deposits at Knossos; these findings are reinforced by work at Phaistos, Crete.⁷ The resultant chronological scheme could offer a possible first step towards the, 'single, shared standardised chronological scheme for the Neolithic Aegean.' However, it would require researchers to adopt a 'common understanding of chronological phasing'. In Anatolia, the multiple phases of the Late Chalcolithic (LCh) span the late 5th millennium to the 3rd millennium BC. Detailed work to refine the chronology and character of the 4th millennium BC in western Anatolia has been the focus of recent work.⁸

In addition to the disparate terminologies, debate continues concerning the best aspects of material culture to investigate the range of transformations associated with the end of the 4th millennium BC – from pottery⁹ (where the question remains as, 'to whether only the study of ceramics together with their context and stratigraphy and the exploration of similarities and differences in the style of material culture are the most appropriate approaches to the investigation of cultural associations, social transformations and changes in the use and meaning of material culture'¹⁰), to settlement patterns, where changes in the second half of the 4th millennium BC are, 'most strongly seen in settlement patterns and social structures' stimulated by outside influence¹¹.

In the study of the evidence for early metallurgy, in particular, there has been a move from provenance-orientated studies, using lead isotope analysis, to small scale site-

⁴ Sampson 1985

⁵ Sampson 1987; 1988 see also Andreou *et al.* 2001

⁶ Tomkins 2007; 2014, 351

⁷ Todaro 2012, Todaro and Di Tonto 2008

⁸ For example, Horejs and Mehofer 2014

⁹ Manning 1995

¹⁰ Mavridis and Tankosić 2016, 419

¹¹ Nowicki 1999; 2002; 2007; 2008

specific archaeometallurgical studies which aim to understand the nature and technology of metallurgical activities in particular contexts¹² There has been criticism of the ‘fetishization’ of the study of metals due to its over-emphasis on production residues and their connection to the trade and environmental sub-system at the expense of causal relationships.¹³ By placing wider societal concerns at the centre of the study of technology [metallurgy], it is possible to address issues such as social identity, or the organisation and control of labour, thereby preventing internalist technological histories.¹⁴ Critics also claim such histories present technological development as the unavoidable consequence of social development - at the expense of obscuring the potential for agent-centred change, and propose studying the varying biographies of different classes of materials and their patterns of consumption to explore the relationships or ‘touching points’ between different aspects of material culture. Such approaches may provide an effective means to understand the organisation of production, process of innovation and context of emergence of metallurgy in the Aegean. Tomkins suggests a practice-based approach as a means of, ‘unpacking the residues of the archaeological record into more meaningful people-thing associations’ This would provide a framework for understanding change in small scale Neolithic societies as it affords an opportunity to, ‘glimpse actual identities’.¹⁵

Spatial

The island of Yali lies off the west Anatolian coast, between Kos and Nissyros and is part of the Dodecanese. The initial occupation of Yali reflects expansion into more marginal environments – a trend observed differentially in the entire Aegean region: the Cyclades islands¹⁶; western Anatolia¹⁷; the Greek mainland¹⁸; Crete¹⁹, and further north, as in Bulgaria (Karanovo III)²⁰. This process must be, ‘assessed contextually and relative to different forms of land use, levels of intra- and inter-

¹² Gale and Stos-Gale 2002; 2003, for example, Georgakopoulou 2011

¹³ Doonan and Day 2007, 4-6

¹⁴ Hughes 1986

¹⁵ 2014, 348, 349

¹⁶ Cherry 1981; 1990 Broodbank 2000 and Davies 2001

¹⁷ For example, at Beycesultan, Kuruçay and Kum Tepe, During 2011 and Horejs and Mehofer 2014

¹⁸ Sampson *et al.* 1999, Mavridis 2006, Georgiadis 2010 and the move to occupy some offshore islands, such as Kephala (Kea), Coleman 1977 and Ftelia (Mykonos) Sampson 2002

¹⁹ Nowicki (2002; 2008), at Knossos, Tomkins 2007; 2008, Kephala Petras, Papadatos 2008

²⁰ Todorova 1999

regional integration, and so on.²¹ Movement into areas less suited to grain production meant greater dependence on stock rearing. This is characterized by seasonality, the need for households to work together, emphasis on storage and a diversification by interaction with neighbouring communities.²²

In the second half of the 4th millennium BC, the changes have three apparent phases – initial occupation of marginal lands (often seasonally worked), then settlement to higher lands or caves (possibly suggesting a security issue). The cause could relate to climate²³, or socio-cultural pressures.²⁴ Marginal occupation is often short term and followed by abandonment.

The occupation and later abandonment of Yali reflects these trends. Survey and excavation suggest Yali was probably seasonally occupied earlier in the 4th millennium, for example at site 2 Laimos on the coast²⁵. Later in the 4th millennium BC, a change occurred - the north-west part of the island became the focus of habitation which was, ‘organised and not accidental.’²⁶ Overall, the higher lands were occupied for longer periods of time, mainly during winter, when cattle were grazed. This may suggest a change in the economic strategies of the islanders or the need to move from sea level. One associated cemetery was identified; others may exist. It testifies to long occupation. Unfortunately, the acidic pumice environment has destroyed much of the cemetery’s contents. Yali was abandoned by the end of the 4th millennium BC.

The specific depositional context for the evidence for metalworking is securely in the mid-4th millennium BC. It was found in a settlement context, in sector Z, which is located on a higher plateau in the north-west part of the island and extends over an area of 1.5 hectares, FIGURE 3. The presence of some earlier pottery, but no structures, suggests some use of this plateau before the mid- 4th millennium BC. However, the foundations of several walls and one building foundation (Z3), of more robust construction, date to after the mid-4th millennium BC. Survey shows that the structures in this sector are of the main settlement on the island at this time; the settlement pattern is complete with smaller outlying homesteads scattered throughout the island.

²¹ Halstead 2008, 233

²² *ibid.* 234

²³ Clare and Weniger 2008

²⁴ Nowicki 2008

²⁵ Sampson 2008

²⁶ Sampson 1988, 257

What can be determined about the nature of this settlement from the remains? The walls of the buildings were mainly constructed in a similar manner; the natural sandstone bedrock was often smoothed to form the floor which acted as the foundation for the walls, (e.g. in Z3, Z6 and Z7). Quarried sandstone from the vicinity formed the lower levels of the walls; smaller pebbles were used for the upper levels. It is difficult to assess the range of building sizes; there were several walls pertaining to different parts of buildings; those on the slopes being the least well preserved. Open spaces are noted between the various walls (buildings).

The crucibles were found in trench Z2 with a great many predominately unpainted coarse ware fragments, mainly large sherds with handles on the rim, some with handles on the shoulder, part of a closed pot and cheese-pots.²⁷ The assemblage was dated by the presence of cheese-pots. In addition, a piece of Melian obsidian, a fishbone, a piece of crab shell and a large millstone were directly associated with the metalworking evidence. On the surface between trenches Z1 and Z2, much ceramic and stone material was found which mirrors that found in the settlement.

The more complete foundations of a building in Z3 may suggest the type of buildings that occupied this elevated plateau²⁸, FIGURE 4. Its walls, preserved to 0,40m, rested on natural sandstone rock. A robust central wall suggests an A-shaped roof. The building has two main rooms and was dug to four levels. In the SE corner of the first room, a raised platform with evidence of burning (hearth) and coarse ware cooking vessels clearly suggests an area for food preparation.

There is evidence that walls (curved in shape) were added to create storage space, supported by the presence of large coarse ware pieces; an alternative interpretation is that it acted as an animal pen. Similar curved walls are noted elsewhere in the settlement: e.g. Trench Z4 and Z13. Given their rough construction, the link between this and later apsidal buildings elsewhere is perhaps fortuitous.

Certainly, the activities apparent in building Z3 appear representative of those on the plateau and elsewhere on the island. From the assemblage, the range of economic activities include: grain production (millstones); animal husbandry (determined from pens and nearby cave use²⁹ rather than bones which were destroyed by the acid

²⁷ Sampson 1988, 42

²⁸ Sampson 1988, 45-54, 57, figure 16a

²⁹ Sampson 1988, 61

environment); milk processing (cheese-pots); fishing (bones) and weaving (pottery impressions). Storage, in the form of large coarse ware pots is testified. Evidence for craft skills in obsidian, pottery and metallurgy all suggest specialisation and also engagement in external contact to procure supplies. Local and Melian obsidian was worked— though apparently differentially.³⁰

The actual context of use is more difficult to ascertain. The crucibles were found alongside a plethora of coarse ware and millstones which are noted throughout the settlement. They are thus located within the domestic environment of the settlement, not separate from it.

In summary, the metalsmiths on Yali worked within the main settlement on the island in the mid-4th millennium BC. The community had a mixed economy and had a range of craft specialists. It is not possible to assess the status of the metalsmith from the evidence.

Typology

The typology of the crucibles is now reviewed to see what light it may shed on the direction of contact or influence.

Though very rare, the finds from Yali contribute significantly to the growing body of evidence for early metalworking. More attention has recently been given to the potential evidence for metalwork in excavations. This has led to improved detection, ‘in the past two decades, excavation of all Greek sites...has found evidence of metallurgy when modern methods of recovery have been employed’.³¹ Whether the increase reflects a greater intensity of research, or better recovery techniques, or not, the finds may represent the ‘tip of the iceberg.’³²

The character of the two coarse ware crucibles, found with copper adhering, are assessed within the local pottery tradition and compared to contemporary evidence in terms of manufacture, design and capacity, FIGURE 5.

³⁰ Georgiadis 2008

³¹ Zachos 2007, 179

³² Sherratt 2007, 248

Manufacture

To prevent distortion and withstand the temperature required for metalworking, the potter must ensure the refractory quality of the clay fabric of the crucibles. Porosity and inclusions in the heat transfer from ceramic to environment, and of temperature development on the entire surface, are most significant.³³ The characteristic voids in the wall structure, reminiscent of burnt out organic temper, was observed on visual inspection of the Yali crucibles. An awareness of specialised clay ‘recipes’ for metalworking operations has been previously suggested.³⁴

Crushed shell, ground obsidian (local) and pumice were used as temper in Yali ceramics; seven ceramics ‘recipes’ were identified, including one where non-local dacite-andesites were added to local Yali clay, representing an intentional technological choice by the potter, which would have improved wall strength and temperature resistance.³⁵ Some suggest that Chalcolithic crucibles were generally made from clay lacking in refractory properties, though the early use of refractory materials has been identified in the crucible from fourth millennium Tepe Hissar, north-eastern Iran.³⁶

The use of such temper is clearly related to the needs of metallurgy; the deliberate production of refractory clays on Yali is confirmed – the crucibles were locally made to meet the needs of metallurgy.

Design

In addition to a deliberate production of clay fabric, the design of each Yali crucible was highly functional. The walls of both become increasingly thick towards the base. Neither base was flat, though B was more even than A. Both crucibles were designed to be placed directly on the heat source, as scorch marks on the bases suggest. This was facilitated by the insertion of a haft into their integral sockets, the thicker bases allowing for this. Essentially, the socket necks are similar in shape; their size simply proportionate to the overall circumference and weight of each crucible. In A, the socket is more rectangular; in B, squarer. The socket direction suggests the crucibles were inserted horizontally – whether at ground or raised level. Heating crucibles from above (blowpipes?) and below (heat source?) can reduce the temperature

³³ Noll 1991, Hein and Kilikoglou 2007, 878-884

³⁴ Blitzer 1995, 504-505

³⁵ Katsarou *et al.* 2002

³⁶ Thornton and Rehren 2009, 2700-2712

difference between the internal and external surface and this reduces thermal stress on the crucible wall.³⁷ The rims are rounded off; the thickness of the rims was around 1.0 cm. Depressions or thinning of part of the rims created a lip or spout to facilitate pouring the molten metal. The lip of crucible A is partially destroyed.

The closest contemporary parallels, for both socketed and non-socketed crucibles are known from the north Aegean, at Thermi III on Lesbos,³⁸ Chalandriani on Syros,³⁹ and on the Greek mainland, in the north, at Sitagroi III,⁴⁰ Sesklo,⁴¹ Mandalo,⁴² and possibly Petromagoula.⁴³ To the south, parallels are known from Alepotrypa⁴⁴ and, in Crete, from Aghia Photia⁴⁵

There is no evidence that different crucible styles imply different metalworking technology, though the socketed variety suggests the need to remove the crucible at a specific point in the process. The two types coexist, in early metalworking traditions at this time; both styles were used for melting/smelting. The non-socketed crucibles produced lozenge-shaped buns of melted/smelted copper; the socketed crucibles produced plano-convex buns. Both styles are known at Thermi, the lozenge style, possibly dating to before Thermi I⁴⁶ and the plano-convex style in Thermi III⁴⁷

Secure handling was necessary to extract the crucibles from the heat, given the weight and molten state of the copper. There is no evidence for casting; it is implied, and moulds would have been of sand, wood, ceramic or stone.

Lack of evidence for crucible lids (clay or wood), if used, may be fortuitous. Lids would have preserved the temperature of the molten copper longer and protected the copper from debris or charcoal. Given the need for visual control, they may only have been used after the melt/smelt.

³⁷ Evely *et al.* 2012, 13

³⁸ Lamb 1936, pl. XXIV, 30, 71

³⁹ Bossert 1967, Fig. 3, 5 and Day 1998, 136

⁴⁰ Renfrew and Slater 2003

⁴¹ Tsountas 1908

⁴² Papaefthimiou and Papasteriou 1997, 1209, 1213, fig. 2

⁴³ Andreou *et al.* 2001, 271

⁴⁴ Papathanassopoulos 1996

⁴⁵ Betancourt and Muhly 2007, 147

⁴⁶ Lamb 1936, pl. XXIV, 30, 43

⁴⁷ Lamb 1936, pl. XXIV, 31, 71

The Yali crucibles are amongst the earlier examples of socketed crucibles in the Aegean. The closest parallels stylistically are from the north west Anatolian littoral.

Capacity

Both crucible A (large) and B (small) are hemispherical with diameters of c. 9 centimetres and c. 3 centimetres, respectively. While not equally symmetrical, the diameter to depth in each is approximately 3:1. The combined volume is roughly 42.4 cms³, or c. 300 grs of copper. There is no reason to suppose that the crucibles were only used once, nor always simultaneously. The difference in size suggests the intention to separate the copper or control quantities. The amount of copper in the Thermi III crucible, in contrast, would have been c. 85-113grs.⁴⁸

Depending on the availability and quality of oxidized copper, and given that the average copper ore yield today is 0.29-2.29%, primitive techniques would probably obtain c. 1.0%. Thus, before beneficiation, at least one hundred times more copper was mined to render 300 grs of copper in the crucibles.

The crucibles suggest the intentional production of small tools or jewelry. The scale of production appears small and is in keeping with other evidence for the period.

Copper

How the copper from the crucibles is situated in the context of the evidence for metalworking in the Aegean is now briefly reviewed.

Though still relatively rare, early evidence for copper artefacts and copper working is more plentiful in central and eastern Anatolia than western Anatolia and the Aegean.⁴⁹ From this study, the earliest finds relatively close to the east Aegean consists of two copper beads from Haçılar (Ia-II) dating to around 6000 BC, though the original place of production is uncertain⁵⁰. Other early evidence also comes from an easterly direction - the copper objects and a crucible from Orman Fidanlığı, dating to the late fifth millennium BC⁵¹; weapons from the cemetery at north central Anatolia at Ilipinar (Phase IV), dating to the first half of the fourth millennium BC,⁵²

⁴⁸ Lamb 1936, Branigan 1974, 71

⁴⁹ Mehofer 2014, 469, Fig. 4

⁵⁰ Yalçın 2000, 19, tab 2

⁵¹ Ay-Efe 2001, 139, 157, pl. 3d-e, Zimmermann 2011, 300

⁵² Roodenberg 2008, 329, figs. 8.5-7; 10.6-7; 12.6-8

and a flat axe dates c. 3,800 BC from Barçın Höyük, in the same area,⁵³ though shown not to be made of local ores.⁵⁴ Crucible fragments and slag are well-attested at Çamlıbel Tarlası in north-central Anatolia dating towards the mid- 4th millennium BC.⁵⁵

South-west of the central Anatolian plain, the earliest finds, after the Haçılar Ia-II finds, come in the form of copper artefacts and crucibles from Beycesultan (level XXXIV)⁵⁶ and Kuruçay, (layer 6A, 6, 4),⁵⁷ with metalworking paraphernalia and copper artefacts known further south from Bağbaşı.⁵⁸ Along the west Anatolian coast, come finds from Bakla Tepe,⁵⁹ Liman Tepe,⁶⁰ Çukuriçi Höyük⁶¹ and, though slightly further north, from Yeşiltepe (but in an unsecured context). A fragment of a ring is also known from Emborio (Chios), layer IX-VIII.⁶² Collectively, these sites rendered pipes, crucible fragments, furnace remains, slags and metal artefacts, all dating to the second half of the fourth millennium BC and thus roughly contemporary with the Yali material.⁶³

In the Greek mainland and west Aegean, the earliest finds of copper and gold artefacts come from Dimitra (Thessaly).⁶⁴ Fourth millennium BC finds on the Greek mainland are known further the north, as ‘by-products’ of copper working at Sitagroi,⁶⁵ with slightly later examples (c.4800-c.4500) from Mandalo,⁶⁶ where slag from a crucible found at Mandalo is reported to contain tin and tin bronze,⁶⁷

⁵³ Gerritsen 2010, 198, 224, fig. 2

⁵⁴ Mehofer 2014, 470, footnote 42

⁵⁵ Schoop 2011; 2015, 47-68

⁵⁶ Lloyd-Mellaart 1962, 19, 21, 112

⁵⁷ Duru 1994; 1996, 56, 125, pls. 159-161, Zimmermann 2011, 300-301

⁵⁸ Erkanal 2008, 168

⁵⁹ Kaptan 2008

⁶⁰ Keskin 2011, 145

⁶¹ Mehofer 2011, 471, Horejs 2014

⁶² Hood 1981, 657, 664, fig. 295.17

⁶³ Mehofer 2014, 471

⁶⁴ Grammenos 1984, pl. 56

⁶⁵ McGeehan Liritzis and Gale 1988, Renfrew and Slater 2003

⁶⁶ Kotsakis *et al.* 1989, 682-30

⁶⁷ Papaefthimiou-Papanthimou and Pilali-Papaseteriou 1997, 146

Dikili Tash,⁶⁸ Limenaria (Thasos),⁶⁹ Mikrothives,⁷⁰ and Palioskala.⁷¹ A little further south, copper finds from Makriyalos are reported⁷². From Thessaly/central Greece, a copper and a gold artefact are known from Dimini,⁷³ with copper from Sesklo,⁷⁴ Pevkakia,⁷⁵ and possibly Mesolonghi.⁷⁶ A copper awl from Aghia Triada, Euboea⁷⁷ and recent finds from Sarakenos Cave⁷⁸, Boeotia add to this list.

From southern Greece, though in less secure contexts, axes from Athens, Euboea and Marathon⁷⁹, compliment the more secure finds Aghia Irini, Kephala copper and copper product from Kephala, (Kea) ⁸⁰and Kitsos cave⁸¹ with evidence for copper exploitation at Spata in Attica,⁸² Koropi,⁸³ with smelting crucibles from the same site,⁸⁴ and Merenda.⁸⁵ In the Peloponnese, the disputed finds from Alepotrypa, of copper, silver and gold, may date to this time.⁸⁶ The same may be true of the axe from Spata,⁸⁷ and copper artefacts from possibly Aghios Dimitrios, Peloponnese.⁸⁸

In the Aegean, finds date to c. 5300-4800 BC, and come in the form of small copper artefacts, copper product and a gold artefact from Ftelia, (Mykonos).⁸⁹ Roughly contemporary with the finds from Yali are copper and gold artefacts from Zas

⁶⁸ Seferiades 1992, 115

⁶⁹ Nerantzis *et al.* 2016

⁷⁰ Adrymi-Sismani 2007

⁷¹ Toufexis 2016

⁷² Pappa and Bessios 1999, 117

⁷³ Tsountas 1908, 354, Volos Museum no. 2364 and 2565

⁷⁴ Tsountas 1908, pl. 4.4 and 4.5, Figure 291-3

⁷⁵ Theocharis 1973, Fig. 125

⁷⁶ Phelps 1979, Branigan 1974, McGeehan Liritzis 1996 and Zachos 2007; 2010

⁷⁷ Mavridis and Tankosić 2016, 429

⁷⁸ Currently being studied by VM

⁷⁹ Phelps *et al.* 1979

⁸⁰ Coleman 1977, 108, Appendix 1, Conophagos 1977

⁸¹ Lambert 1981

⁸² Zachos 2010, figs. 6-4

⁸³ Kakavoyiannis *et al.* 2009

⁸⁴ Amzallag 2009

⁸⁵ Kakavoyiannis *et al.* 2008

⁸⁶ Papathanassopoulos 1996

⁸⁷ Phelps *et al.* 1979

⁸⁸ Zachos 1987, 81-82

⁸⁹ Sampson 2002, Maxwell *et al.* 2018, 155

(Naxos),⁹⁰ and copper from Strofilas (Andros).⁹¹ There is also a copper axe from Knossos,⁹² evidence for copper working from Kephala Petras (Crete),⁹³ and possibly contemporary copper artefacts Chrysokamino.⁹⁴

Overall, copper was worked and used on both sides of the Aegean at the same time as on Yali and the general character is one of varied technological preferences and practices in a context of high mobility.

Lead

How the lead rivet from Yali is situated in the context of evidence for lead (and silver) working in the Aegean is now briefly reviewed.

One lead rivet was discovered at the settlement on Yali in association with the ceramic crucibles. No evidence for lead working was found, though, 'if lead is found in early archaeological contexts, it is by itself evidence for the smelting of ores'.⁹⁵ Extraction of lead silver and copper from Siphnos began in the 4th millennium⁹⁶ and also on Kythnos.⁹⁷

The presence of the rivet confirms lead could be acquired. Its use as a rivet suggests it was a low value item - finer pots were obviously considered of more value, materially, aesthetically or culturally.

The lead from Bakla Tepe⁹⁸ and the silver from Beyçesultan⁹⁹ are the closest relevant geographical and chronological artefacts here. The closest example for copper and lead working comes from Çukuriçi Höyük in the fourth millennium BC.¹⁰⁰ Indeed, the copper silver alloy at Çukuriçi Höyük is taken to fill the gap between the

⁹⁰ Zachos 1987

⁹¹ Televantou 2008

⁹² Evans 1964

⁹³ Papadatos 2007, 154

⁹⁴ Betancourt 2006, 57

⁹⁵ Pernicka 2014, 448

⁹⁶ Wagner and Weisgerber 1985, Gropengiesser 1987

⁹⁷ Hadjianastasiou and MacGillivray 1988, Stos-Gale 1989, Hadjianastasiou 1998 and Papadatos 2007

⁹⁸ Şahoglu 2014, 17

⁹⁹ Lloyd and Mellaart 1962, 280-283

¹⁰⁰ Horejs 2014, 23, Mehofer 2014, 463-90

appearance of this alloy in the Balkans and eastern Anatolia.¹⁰¹ Both lead and copper from Çukuriçi Höyük has the same lead isotope (thus, geological) age. Analysis may show that the same is true for the copper and lead from Yali.

Overall, evidence for copper and lead working exists in the Aegean region from the middle of the fourth millennium BC. Evidence for each increases in the Early Bronze Age.¹⁰² The finds from Yali confirm the island had access to copper and lead; there is increasing evidence for polymetallism in the fourth millennium BC.¹⁰³

Analyses

The chemical analysis of the copper from both crucibles and lead isotope analysis (LIA) of the coppers and the lead are presented here. The chemical reanalysis is justified to help resolve the question of possible early presence of tin in earlier analysis of the copper.

Chemical Analysis

To determine the technological nature of metalwork at the site, the oxidised copper product from both crucibles underwent Inductively Coupled Plasma- Optical Emission Spectroscopy (ICP-OES) by Professor R. M. Ellam of the Scottish Universities Environmental Research Centre. The methodology is described in Appendix I (a). Previous Atomic Absorption Analysis (AAS) of the copper from crucible A, by Dr. K Assimenos of the National Museum of Athens in the 1980's had identified traces of tin in the copper, FIGURE 6. In the AAS results, the analytical total for the corroded/oxidised copper in crucible A is under 22.5%. When normalised, to suggest the plausible composition, the errors are enormous. Warnings have been given regarding extrapolation from such low percentages.¹⁰⁴

The AAS results suggested the presence of arsenic and tin in the copper. Arsenical copper with tin is very rare, especially at this time – certainly atypical of the Anatolian metal industry generally.¹⁰⁵ As any use of tin indicates a technological watershed, the copper was re-examined. The issue of tin is reviewed further in Appendix II.

¹⁰¹ Mehofer 2014, 467, footnote 20

¹⁰² Georgakopoulou 2013

¹⁰³ Moorey 1994

¹⁰⁴ Craddock 1980

¹⁰⁵ De Jesus 1980, Wagner *et al.* 1986, Craddock and Meeks 1987, Palmieri *et al.* 1992 and Özbal *et al.* 2000

The ICP-OES results: where elements were detected, or looked for, quantified concentrations are provided: the original, pre-normalised, results - the raw intensity - data and the normalised data are both given in FIGURE 7. Given the much higher percentage of copper determined, extrapolation of constituent elements is more valid, though previous warnings must still be considered.

As there were no standards, much of the shortfall in the heavily oxidised copper for Yali 2 (Crucible A), 71.3% analysed, appears to be Fe and Mn whereas for Yali 3, (Crucible B), the copper, though still oxidised, is purer at 95.6% with much lower Fe. The arsenic levels suggest the use of a copper deposit which naturally contained arsenic.¹⁰⁶ Some contend that intentional arsenical copper alloys may have resulted from the use of an iron arsenide,¹⁰⁷ as early as the EMI at Poros Katsambas. Others suggest the use of the speiss to explain the presence of arsenic in copper artefacts from Çukuriçi Höyük.¹⁰⁸ The appearance of low arsenic in early copper is indicative of the use of a copper source with naturally occurring low arsenic content which is the most probable explanation for the Yali copper.

To ascertain if the copper was itself the product of smelting in an earlier phase of its biography, the chemical criteria for minor/trace elements would have to be observed. However, the state of preservation does not really allow this. What can be said about the nature of the copper used on Yali is that the copper was pure, it contained traces of arsenic natural to the source. No tin was found in the reanalysis.

Lead Isotope Analysis

The copper and lead from Yali underwent lead isotope analysis to ascertain the geological age of the material used to make them. This is then related to sources of lead and copper for which data exists. Not all potential sources have been analysed or characterised in this way. Issues with the method and the interpretation of results persist and are reviewed briefly here prior to the discussion of the results themselves.

Even without the complex ore geology of Greece and the Aegean, there are limits to lead isotope analysis on its own. The issue of provenance and issue of mixing of

¹⁰⁶ McGeehan Liritzis and Gale 1988

¹⁰⁷ Doonan *et al.* 2007, 113

¹⁰⁸ Mehofer 2014, 467-468

coppers has engendered much debate.¹⁰⁹ Mineralogical work and progress in the resolution of ore fields are significant in encouraging confidence in interpretation of results.¹¹⁰

The best way to plot, interpret and discuss representations of lead isotope analyses also remains contentious, as a comparison of arguments will confirm.¹¹¹ Archaeologists come under fire for not understanding the limits of the method; scientists come under fire for not always fully appreciating the archaeological implications. Knapp¹¹² argues that lead isotope analyses can never be used to assign specific provenance to artefacts, only to exclude certain provenances. However, Gale and Stos Gale¹¹³ justify the use of their results in this way. They quote the work by the originators of the technique¹¹⁴ as well as the practice of other laboratories that use the exclusion principle¹¹⁵

What is the possibility that local sources exist on Yali? Even though Yali is placed within the Attic-Cycladic belt, its geology does not support the presence of copper or lead ores. Within the Aegean, successive stages of the tectonic and metallogenetic evolution of the Attic- Cycladic belt during the Miocene has resulted in the formation of numerous lead-silver-zinc-copper and gold deposits and mineral occurrences.¹¹⁶ Dispersions of secondary copper minerals, like malachite and azurite, can be seen in Laurion, Siphnos, Kythnos, Kalianou (south-east Euboea) and Kea. They were sites of intense exploitation in ancient times.

There are possible ore occurrences on Kos, where chalcopyrite is reported for Dikaion Mountain. Copper deposits are known from Serifos, at Auyssalos and Kephala¹¹⁷ and there is archaeometallurgical evidence on Syros at Chalandriani and Kastri though exploitation of the source has not yet been proved.¹¹⁸ Sources near

¹⁰⁹ For example, Gale and Stos Gale 1992; 1993; 1996, Sayre *et al.* 1992, Pernicka 1992; 1995 and Budd *et al.* 1993; 1995; 1996; Budd and Taylor 1995

¹¹⁰ For example, Stos Gale *et al.* 1995 and Sayre *et al.* 2001

¹¹¹ Knapp 2002, Gale 2003

¹¹² 2002, 40

¹¹³ 2000, 522-535 and Gale 2003, 60

¹¹⁴ Brill and Wampler 1967, Grögler *et al.* 1966

¹¹⁵ For example, Pernicka and Wagner 1982, Farquhar and Fletcher 1984, Pinarelli *et al.* 1995, Hauptmann *et al.* 1999; 2003 and Sayre *et al.* 2001

¹¹⁶ Skarpelis 2002

¹¹⁷ Salemink 1985

¹¹⁸ Skarpelis and Triantafylidis 2004

Bakla Tepe in the Izmir region have been verified¹¹⁹ A local polymetallic source is known near Çukuriçi Höyük¹²⁰ and it may have supplied that local industry.¹²¹ Copper is known at Kalithies on Rhodes where a copper chisel dating to the fifth millennium BC is known from nearby.¹²²

The copper and lead used on Yali was not local; they could have come from sources on either side of the Aegean. The copper would reach the island in the form of pre-smelted copper, beneficiated copper mineral or possibly even as a previous artefact. Comparing the lead isotope results for the Yali copper and lead and the geological and lead isotope data currently available for sources in the region may help ascertain the most likely source which supplied the islanders.

Two sets of lead isotope results for the copper are available for the Yali coppers. These are the earlier determinations of the copper product from both crucibles by Professor N.H. Gale, Oxford University, dating to the 1990's, FIGURE 8 and for this paper, determinations on further copper samples from each crucible, in addition to the lead rivet by Professor R. M. Ellam, FIGURE 9.

The method used to determine and interpret the lead isotope data at SUERC is the same as that used at Oxford.¹²³ The specific procedure followed at SUERC is outlined in Appendix I (b).

The LIA confirm the close geological age of the copper from both crucibles and the lead rivet which suggests exploitation, originally, of one source which was polymetallic in character. There are parallels for this on both sides of the Aegean.

The LIA determinations for Yali were compared with current, yet incomplete, lead isotope data on Greek, Aegean, Balkan and Anatolian sources, mainly catalogued in OXALID¹²⁴, together with mineralogical data on sources or contemporary artefacts, FIGURE 10.

¹¹⁹ For example, Şahoglu 2008; 2014, 71

¹²⁰ Horejs 2010, Mehofer 2014

¹²¹ Mehofer 2014, 466

¹²² Sampson 1988, 219

¹²³ Stos-Gale *et al.* 1995, Gale and Stos-Gale 2000 and Stos-Gale and Gale 2009

¹²⁴ <http://oxalid.arch.ox.ac.uk/> and references therein; Stos-Gale and Gale 2009

The north-west Anatolian field has been determined.¹²⁵ These sources have been identified as polymetallic, in copper and lead. Though generally discrete, the southern extent of the field does overlap in part with the Greek mainland source at Laurion (also polymetallic in copper, lead and silver). Early evidence that north-western sources were accessed early by communities in north-west Anatolia comes from Beşiktepe, Poliochni and Thermi.¹²⁶ Copper sources further south along the central Anatolian coast, in the Izmir region, are known and copper working is verified there in the Late Chalcolithic, in the lead and copper finds from Bakla Tepe¹²⁷ where pending analyses may help clarify sources accessed.¹²⁸ Recent lead isotope and chemical results for material dating to the end of the long Late Chalcolithic phase (start of the 3rd millennium BC) at Çukuriçi Höyük confirm that it accessed north-western Anatolian sources, or sources of a similar geological age¹²⁹

Çukuriçi Höyük is the closest metalworking site geographically to Yali, though it had access to a different source for copper and lead. While the copper from crucible A and B does overlap slightly with the southern extent of the north-west Anatolian ‘field’ (which is less precise), they plot distinct from the Çukuriçi Höyük material which rests more firmly within that field.¹³⁰ Further, the mineralogical character of the finds from Çukuriçi Höyük is smelted copper with higher arsenic (use of speiss suggested).¹³¹ The closest artefactual evidence to Yali comes from Beyçesultan, phase XXXIV where copper and a crucible are known.¹³² However, lead isotope results for this material is not currently available.

The Yali results are now compared to known Aegean and Greek mainland sources.¹³³ It is apparent that there is a slight difference between the determinations of Gale and Ellam when plotted. Gale’s copper from crucible A and B rest on the border of the Laurion and Kythian ‘fields’ - as does Ellam’s result for crucible B copper. The result for the copper from crucible A and the lead rivet fall more firmly within the

¹²⁵ Begemann *et al.* 1994, 204, 193, table 4, Pernicka *et al.* 1997, Begemann *et al.* 2003, 193, table 4, Gerritsen *et al.* 2011, 212, tab 2

¹²⁶ Pernicka *et al.* 1990, Gale *et al.* 1984 and Stos Gale 1992

¹²⁷ Şahoglu 2008; 2014

¹²⁸ Şahoglu and Tuncel 2014, 71, footnote 22

¹²⁹ Mehofer 2014, 474-7

¹³⁰ Mehofer 2014, 474-7, Figs. 7-9

¹³¹ Mehofer 2014, 465-8, footnote 20

¹³² Lloyd Mellaart 1962, 19, 21, 112

¹³³ Stos Gale *et al.* 1995

Kythnian field which was known to have been exploited.¹³⁴ Given the possibility of some overlap at the southern end of the north-west Anatolian field, it is technically possible that the copper and lead found at Yali could have come from either side of the Aegean – the most convincing probability is that the copper came from Kythnos, or from a polymetallic source with a similar geological age which is currently not known geographically. The Balkan sources are quite distinct. The Yali metals are distinct from the copper and lead used at Çukuriçi Höyük. This is perhaps the earliest evidence for the exploitation of the source at Kythnos.

If indeed the community on Yali acquired its copper from the west Aegean, mobility is a key characteristic in raw materials acquisition and knowledge exchange at the end of the Neolithic. On current evidence, Laurion copper reached as far north as Sitagroi (Macedonia) and Dimini (Thessaly) in the fourth millennium BC.¹³⁵ In the south, it seems to have been accessible to early 4th millennium BC communities on Kephala (Kea)¹³⁶ later 4th millennium BC Zas (Naxos)¹³⁷ and, amongst other sources, early in the 5th millennium BC on Ftelia (Mykonos).¹³⁸ Further, several communities involved in metalwork at the end of the Neolithic are known to have had access to more than one source of copper: Sitagroi accessed three; Dimini accessed two, Sesklo accessed at least two,¹³⁹ Zas accessed two,¹⁴⁰ and Ftelia accessed at least three.¹⁴¹

Overall, no copper source is known to exist on Yali; the copper and lead were imported, and they came from the same, polymetallic source. There are sources close to Yali on the Anatolian littoral and the Dodecanese, yet Kythnos appears to be the most probable source and the results from Yali confirm its early distribution in the east Aegean. Procuring metals from a distance was facilitated by developments in sea transport and this suggests metallurgical emergence in circumstances of high mobility. Variety in the Aegean at this time suggests variable technological preferences and practices in the mid-4th millennium BC.

¹³⁴ Hadjianastasiou and MacGillivray 1988, Stos-Gale 1989, Hadjianastasiou 1988 and Bassiakos and Philaniotou 2007

¹³⁵ McGeehan Liritzis and Gale 1988, 210-211

¹³⁶ Coleman 1977, 108

¹³⁷ Zachos 1987

¹³⁸ Maxwell *et al.* 2018

¹³⁹ McGeehan Liritzis and Gale 1988, 210

¹⁴⁰ Zachos 2007, 173

¹⁴¹ Maxwell *et al.* 2018

Conclusions

Yali is geographically located in a region of the Aegean active in metalworking at the end of the 4th millennium BC. The evidence for metalworking was found in the main settlement on the island at this time – a settlement practicing a range of crafts underpinned by a mixed economy.

The crucibles demonstrate cultural links with the east Aegean. The copper and lead come from the same source (Kythnos) confirming links with the west Aegean. This is evidence for the early use of the Kythnos source. There is only one verifiable instance of copper smelting in the FN, Kephala (Kea)¹⁴² The metals were imported and transported by sea. There is growing evidence for a range of sea transport at this time.¹⁴³ The longboat is verified from the end of the 4th millennium BC.¹⁴⁴ and may be operating in the Aegean when the community at Yali were trading. involvement in trade or exchange networks active in the Aegean is clearly demonstrated in the acquisition of Melian obsidian and pottery as well as metals. Exactly how procurement was organised is more difficult to assess, but knowledge of where to acquire copper was shared and resources (in labour, time) assigned.

The distribution of copper artefacts and the evidence for metalworking at the end of the Neolithic in the Aegean appears often to bear little or no immediate relationship to the copper resources themselves. Indeed, the inception, reception and exchange of metalworking knowledge appears to have more to do with drivers other than the availability of copper itself and to this end, we should assess issues such as the availability of fuel, water, cultural preferences or individual agency. Involvement was socially driven.

The fact that the crucibles can be situated within the local pottery tradition of the island confirms knowledge sharing between potter and metalsmith, suggesting a possibly link in their social identities. Pyrotechnological skills underpin the technological context of emergence, a ‘touching point’ between two different aspects of material culture. It is difficult to further assess the status of the metalworker from the evidence, however.

There is no evidence to suggest that production was for other than local consumption, though this did not appear to include burial.

¹⁴² Coleman 1977, 108

¹⁴³ McGeehan Liritzis 1988

¹⁴⁴ Televantou 2004

Regarding scale, the preparation of the ceramics of this size implies a preconceived understanding of the amount to be worked and the intention of producing small tools or jewelry. Tools would enhance other craft specialisations on the island – lapidary, pottery, or textiles. Jewelry would have had symbolic or aesthetic significance. It is unlikely that this would be the only example of modification of pottery to suit metallurgy; the crucibles could also have been used repeatedly. Given the context of rescue excavations, it is likely that more evidence for metalworking formerly existed at the site.

A key difference between metalworking in the 4th millennium BC and in the succeeding Early Bronze Age is scale. In assessing the nature and technology of metallurgical activities, in context, in the southern Aegean, Georgakopoulou focused on the issue of how mobility is manifested within the first stages of metal production and also how technological variability and homogeneity present themselves.¹⁴⁵ From this, some parallels between the nature of the FN (as on Yali) and EBA industry in the Aegean can be drawn: evidence for the lack of relationship between source and place where metal is worked (that is, scarcity of metal resources bears little relationship to involvement of communities in metalworking); the ceramics used and metal produced shows much technological variability suggesting a range of different practices coexisting at the same time, and, the clear relationship between mobility and metallurgy. Pryce *et al.* have also shown that, ‘there was much local variation in metallurgical practices in the Early Bronze Age Aegean.’¹⁴⁶ This appears to be the case at the end of the Neolithic, too, despite the claim that there are, otherwise cultural ‘indicators of fourth millennium connectivity’.¹⁴⁷ It appears that these communities across both periods had agency.

Acknowledgements

V.M. is grateful to Professor A. Sampson for permitting the study of the artefacts and to the anonymous referee for detailed comments. To Alex and Katherine, as always.

¹⁴⁵ Georgakopoulou 2016, 1-24

¹⁴⁶ Pryce *et al.* 2007, 553-4

¹⁴⁷ Horejs 2014, 15

APPENDIX I

(a) Methodology – ICP-OES and LIA

Chemical Analyses at SUERC:

ICP-OES Methodology

Sub samples were analysed for a range of metals using Inductively coupled plasma – optical emission spectroscopy (ICP-OES).

To prepare samples for ICP-OES analysis, the established method used in the SUERC ICP-MS/OES laboratory was used. 0.1 g sub-samples of dried, ground homogenised peat were refluxed for 8 hours in 2 ml of an aqua regia solution (Aristar grade 12M HCl: 16M HNO₃, 50:50) in a covered Teflon beaker. The refluxed solutions were then diluted to 10 ml with Milli Q (R=18.2 Mohms.cm@25°C) water and filtered through a Whatman 542 grade hardened ashless filter paper to separate from the peat residue, the residues rinsed with Milli Q water and the combined filtered solutions and rinsings made up to a final volume of 100 ml with Milli Q water in an A -grade volumetric flask. Blanks and a Certified Reference Material, CRM049-050 (metals in soil- RTC, Laramie, WY 82070- used in house for inter-comparison of various soil matrices) were also analysed in the same manner. The resulting solutions were analysed for Pb, Zn, Fe, Mn, Cu, Ni, Cr, Cd, Ca and Mg using a Perkin Elmer 5300DV ICP-OES instrument, with a Scott style spray chamber and gem tip cross flow nebuliser under the conditions in the following Table.

Torch gas flow rate	Auxiliary gas flow rate	Nebuliser Gas flow rate	RF power Watts	Viewing distance	Plasma view	Sample flow
15 l min ⁻¹	0.2 l min ⁻¹	0.8 l min ⁻¹	1300 watts	15mm	Axial	1.5ml min ⁻¹

The spectroscopic lines used for analysis of each element are detailed in the next Table.

Analyte Element	Atomic Spectroscopic Line (nm)
Pb	220.353
Zn	206.200
Fe	238.204
Mn	257.610
Cu	327.393
Ni	231.604
Cr	267.716
Cd	228.802
Ca	317.933
Mg	285.213

Peak area was integrated over 3 points with a 2-point background correction. Calibration of the instrument was carried out using 0.1, 1.0 and 10.0 mg l⁻¹ standards prepared from NIST traceable Alfa Aesar Specpure® 1000 mg l⁻¹ standards for each of the metals under analysis. For quality control purposes the validity of the method was assessed by analysis of a Certified Reference Material- CRM049-050 (Metals on Soil) with results detailed in the following Table.

CRM049-050 Metals on Soil Units: mg kg⁻¹					(n = 4)	
Element	Certified Reference value	Standard Deviation	Confidence Interval	Prediction Interval	Average measured value	Standard Deviation
Pb	111	6.69	109 – 112	97.5 - 124	118	7.12
Zn	542	28.9	534 – 549	485 - 599	616	41.0
Fe	9170	774	8950 - 9380	7630 - 10700	8791	391
Mn	636	37.7	625 – 646	561 - 710	628	33.3
Cu	88.5	5.39	87.1 - 89.8	77.8 - 99.1	83.4	4.87
Ni	344	19.9	339 – 349	304 - 383	349	28.4
Cr	355	20.7	350 – 360	314 - 396	355	13.7
Cd	80.0	4.28	78.9 - 81.0	71.5 - 88.4	80.3	4.25
Ca	4790	392	4680 – 4910	4020 - 5570	5126	682
Mg	899	61.4	881 – 916	777 - 1020	842	38.1

For Mn, Ni, Cr and Cd the average measured value (n=4) for the Certified Reference Material fell within the Confidence Interval range, while for Pb, Fe, Cu, Ca and Mg

the value fell outwith the Confidence Interval range but within the Prediction Interval. For Zn, the value fell outwith both of these ranges suggesting a blank problem.

(b) LIA Methodology at SUERC

Analytical Methods and Instrumentation

The MC-ICP-MS data was determined using the Tl-doping method.¹⁴⁸ Samples were prepared with a single anion column pass to achieve a sufficiently pure Pb separation top yield intense ion beams. Separated samples were diluted to 50ppb Pb in 5% HNO₃, that was doped with 5 ppb of NIST SRM997 Tl.

The Pb isotope compositions were measured on a Micromass IsoProbe MC-ICP-MS using an Elemental Scientific Inc 100 $\mu\text{l min}^{-1}$ PFA nebulizer and Glass Expansion Pty microconcentric spray chamber. 208-Pb beam intensities were of the order of 10 Pa and each measurement consisted of 5 blocks of 20 ratios (5 s integrations) collected in static multi-collection mode. Each measurement consumed about 50 ng of Pb (roughly 10% of the separated Pb for the most Pb-rich samples). Mass bias was corrected using the doped Tl assuming an exponential law and 205-Tl/203Tl=2.3871. Baselines were measured on peak for 45 s in blank 5% HNO₃ prepared with the same Milli-Q 18.2 M Ω water and 2xTeflon sub-boiling distilled concentrated nitric used to dilute samples. A solution of 5% HNO₃/2% HF was introduced into the ICP_MS for 2 min between samples and was found to limit the build-up of Pb memory to <3 Mv at mass 208 during the course of an analytical session of several hours. Several standards were measured during each analytical session and the mean of these used to correct for small inaccuracies using the triple-spiked TIMS data as reference composition (all 2 s.d. N-79). The major cause of inaccuracy is probably due to failure of Tl-normalisation to estimate mass bias of the Pb isotopes. In common with other studies,¹⁴⁹ this phenomenon manifests as decreased accuracy with increasing mean mass of the isotopes forming a particular ratio, e. g. 208Pb/204 is less accurate than 206Pb/204Pb. However, other sources of inaccuracy may include (i) incomplete correction of Hg interference on mass 204 which would arise if molecular species contribute to masses 202 and 201 used to monitor Hg; (ii) differences in Faraday collector efficiency; and (iii), variations in ion

¹⁴⁸ Rehkämper *et al.* 1998, Belshaw *et al.* 1998 and White *et al.* 2000

¹⁴⁹ *Ibid.* 139

energy across the focal plane of the mass spectrometer. The errors quoted in the results (previous Tables) are given with standard errors.¹⁵⁰

Overall, the procedure used at SUERC determine the lead isotope ratios of the copper artefacts, ore and lead ores from Yali follows that undertaken in at Oxford and other laboratories and this permits comparison of results. In each the procedure to plot the results are as follows: firstly, the Euclidean distances in the three-dimensional space with axes defined by the three LI ratios are calculated between each of the artefact's LI ratios and all currently available LI data points for ore and slag samples. Software such as TestEuclid sorts out the data in the order of increasing Euclidean distances. The LIA ratios of the artefact and an ore sample are regarded as identical if all three ratios for both are within the analytical error for the each of the three LI ratios. Next, the geochemical, geographical and historical (archaeological) information is considered. Finally, the data points are compared in two-dimensional graphical plots of LI ratios of the artefacts and ores selected in the previous two steps. The two –dimensional scatter plot was selected over multi-variate statistical analysis of LIA¹⁵¹ in this instance, because it affords comparison with the plethora of earlier data and the results.

APPENDIX II

The Issue of Tin

Clearly, the chemical results (AAS and OES) do not agree on the issue of tin in the copper from the Yali crucibles. In the OES results, where the figures for constituent elements are not readily converted, tin (Sn) was one of the elements that was standardised, so the concentration for Yali 2 (Crucible A) of 118 ppm (0.0118%) and, for Yali 2 Crucible B) of 885 ppm (0.0885%) should be reliable. These percentages are different, but still lower than would be expected if tin had been deliberately added. It does suggest that the copper source had tin minerals associated with it and that the copper from Yali does not represent a copper-tin alloy.

Reviews of the problem of tin availability have concluded that tin, as cassiterite, is rare in the Aegean and alloys made from tin ore come from a later date.¹⁵² Some¹⁵³ assert that since copper and tin minerals rarely occur together, the intentionality of tin bronze is not contentious, and the introduction of tin bronze

¹⁵⁰ To calculate these = $(1SE\%/50) \times \text{the relevant ratio}$ e.g. $18.809 \pm 0.0080\% = 18.809 \pm ((0.0080/50) \times 18.809) = 0.003$. The various ratios are expressed to the same number of decimal places as the errors.

¹⁵¹ As adhered to by Sayre *et al.* 2001

¹⁵² For example, Muhly 1999; 2006

¹⁵³ Doonan *et al.* 2007, 102

thus represents a technological horizon. In this horizon, cassiterite, or tin ore, needs first to be made into a concentrate (by panning or vanning¹⁵⁴). Pronounced segregation can be a problem in the interpretation of small samples.¹⁵⁵ The concentrate is smelted by heating with charcoal. The tin metal is then added to the molten copper oxide, in the furnace or even in the crucible.¹⁵⁶ Allowing for segregation and the absence of iron, is it likely that the tin ore product was knowingly added to copper at Yali? If this technological level was reached, what form did the tin take?

Though rare, tin minerals, just as arsenical minerals, do occur in sectors of copper ore bodies in the Aegean. Based on extensive geological fieldwork,¹⁵⁷ it has been convincingly argued that some potential outcrops of tin mineralisation should be considered as potential tin sources in the Aegean. A ‘tin oxide’ or ‘hydrous tin oxide’ compound, corresponding to the minerals romanarchite and hydromarchite respectively, is produced at the oxidation zone of supplied mineralisations, comprising tin-bearing mineral species of the sulfosalt group (e.g. stannite, keosterite). In epithermal-type ores, copper-arsenic and copper-tin sulfosalts can occur. Epithermal mineralisations are widespread in the Aegean and adjacent areas, being part of a Tertiary metallogenetic belt extending from the southern and north eastern Aegean islands. Localised outcrops are noted on Tinos and Syros in the Cyclades in particular.¹⁵⁸ On Tinos, for example, tin bearing sulfosalts and minor cassiterite has been identified;¹⁵⁹ on Syros, oxidation is pronounced, and stannite has been identified.¹⁶⁰ There are reports¹⁶¹ of tin ores on Skyros similar to that located near Sitagroi (Macedonia): Sitagroi IV had the first instance of tin in copper in the broader Aegean.¹⁶²

The argument presented for the unintentional mixing of tin salts with copper suggests one possible way, possibly accidental way that early very low tin ‘bronzes’ were produced. It has been suggested that stannite may be the source of the tin in low tin bronzes.¹⁶³ Others have noted the presence of stannite near early

¹⁵⁴ Amply described by Yener and Goodway 1992, Yener and Vandiver 1993, Earl and Yener 1995 and Adrians *et al.* 2000

¹⁵⁵ Adrians *et al.* 2000

¹⁵⁶ Earl and Özbal 1996; 2006

¹⁵⁷ Skarpelis 2002

¹⁵⁸ Skarpelis and Triantafylidis 2004

¹⁵⁹ Meledonis 1980, Tombros and Seymour 1998

¹⁶⁰ Meledonis and Constantinides 1983

¹⁶¹ Skarpelis 2007

¹⁶² Skarpelis and Triantafylidis 2004, McGeehan Liritzis and Gale 1988, 220

¹⁶³ Charles 1975, 21

cassiterite.¹⁶⁴ It could enter through a gossan flux.¹⁶⁵ Small traces of tin may be characteristic of some Anatolian copper deposits.¹⁶⁶ As we shall see, none of these are of the same geological age as the copper used on Yali.

Such mineralisation could account for early (low) tin bronzes and this may have led to an increased interest in cassiterite. Arsenic mineralisation may have similarly encouraged interest in arsenic rich copper. The use of arsenide at EBA Poros Katsambas may be yet another example of experimentation.¹⁶⁷ It could represent progression in the use of arsenical coppers in an effort to attain consistency. Overall, however, the presence of tin at this inception stage in metallurgy should not be ignored.

The occurrence of tin in copper in the Aegean at this time is relatively rare but does exist. There is much regional variation. In the north, some examples include the 5.9% and 2.3% percentages of tin in two pieces of copper from Sitagroi IV (EBA I), thought to be the earliest in the Aegean.¹⁶⁸ Contemporary with the Yali finds is the crucible with copper slag and evidence for tin come from phase II at Mandalo, northern Greece.¹⁶⁹ This site had strong ceramic connections with both the Rachmani culture of Thessaly and the Maliq II culture of Albania.¹⁷⁰ The lead isotope analysis of the Mandalo copper plots the source and artefacts into a distinct southern part of the Laurion 'field'.¹⁷¹

In the Balkans, where tin in copper appears later than at Sitagroi, levels ranged from 6-10% and represent the use of tin ore, for example at sites such as Eneolithic Karanovo¹⁷² and Gomolava Pločnik D1 during the Vinča period.¹⁷³

In the east Aegean, a Thermi I pin, with 13.1%,¹⁷⁴ must certainly have been made from tin ore, as would its successor from this site: a tin bangle from Thermi (III?) and a punch from Thermi IV with 1.65% tin.¹⁷⁵ Before this, the Mersin conical

¹⁶⁴ Muhly 1973, 98

¹⁶⁵ Charles 1980, 173

¹⁶⁶ Gale *et al.* 1984, 38

¹⁶⁷ Doonan *et al.* 2007, 112

¹⁶⁸ Slater 1972, McGeehan Liritzis and Gale 1988, 220

¹⁶⁹ Kotsakis *et al.* 1989, Papaefthimiou-Papanthimou and Pilali-Papasteriou 1977, 146

¹⁷⁰ Pilali-Papasteriou *et al.* 1986, 465

¹⁷¹ McGeehan Liritzis 1996, 363, no. 453, analysis 81

¹⁷² Chernykh 1978

¹⁷³ Ottaway 1979

¹⁷⁴ Lamb 1936

¹⁷⁵ Lamb 1936

headed toggle pin, dating to the Late Chalcolithic (c. 4,300 BC), has 1.3% tin and 1.15% arsenic.¹⁷⁶

Closer to Yali, most of the earliest finds are located either in north western or western Anatolia or in the offshore islands of the east Aegean. The quantities of tin imply use of tin ore. *If* one accepts the tin results for Yali, it may be considered an early instance of this group

¹⁷⁶ Garstang 1953, 108, Esin 1967, E-17884 and Yakar 1984, I, 60, note 7

Figures

Figure 1: Yali in the Context of the Aegean, with some key sites noted

Figure 2: Chronological Relationship of Yali to 4th Millennium BC Communities in the Wider Aegean

Figure 3: Spatial Context of Yali – the island › the higher plateau › the location of the main settlement with findspot of the crucibles in Z2, (After Sampson (1988, figures 4 and 9))

Figure 4: The Apsidal Features in a building on Yali, (After Sampson (1988, 54, 16a))

Figure 5: The Crucibles from Yali, A - larger; B – smaller, (After Sampson, 1988, figure 64, 188)

Figure 6: Chemical Composition of Copper in Crucible A (Atomic Absorption Spectroscopy, after Dr. K. Assimenos, National Museum of Athens Laboratory).

Figure 7: Chemical Composition of Copper in Crucibles A and B (Optimal Emission Spectroscopy Results of Two Copper Products from Yali Crucibles (raw data (a) and normalised (b)), after Professor R. M. Ellam, Scottish Universities Research and Reactor Centre).

Figure 8: Lead Isotope Determinations of Copper from Crucibles A and B (after Professor N. H. Gale, Laboratory for Archaeometry, University of Oxford), www.oxalid.ox.ac.uk.

Figure 9: Lead Isotope Determinations of Copper from Crucibles A and B and Lead Rivet, (after Professor R. M. Ellam, Scottish Universities Research and Reactor Centre, University of Glasgow)

Figure 10: Lead Isotope Results, (after Gale and Ellam. Samples 1 and 2 = Gale's analyses for Crucible A and B; Samples 3, 4 and 5 = Ellam's analyses for Crucible A and B and lead rivet, respectively, as they relate to the Kythnos field (determined by Gale and Stos Gale (1986)).

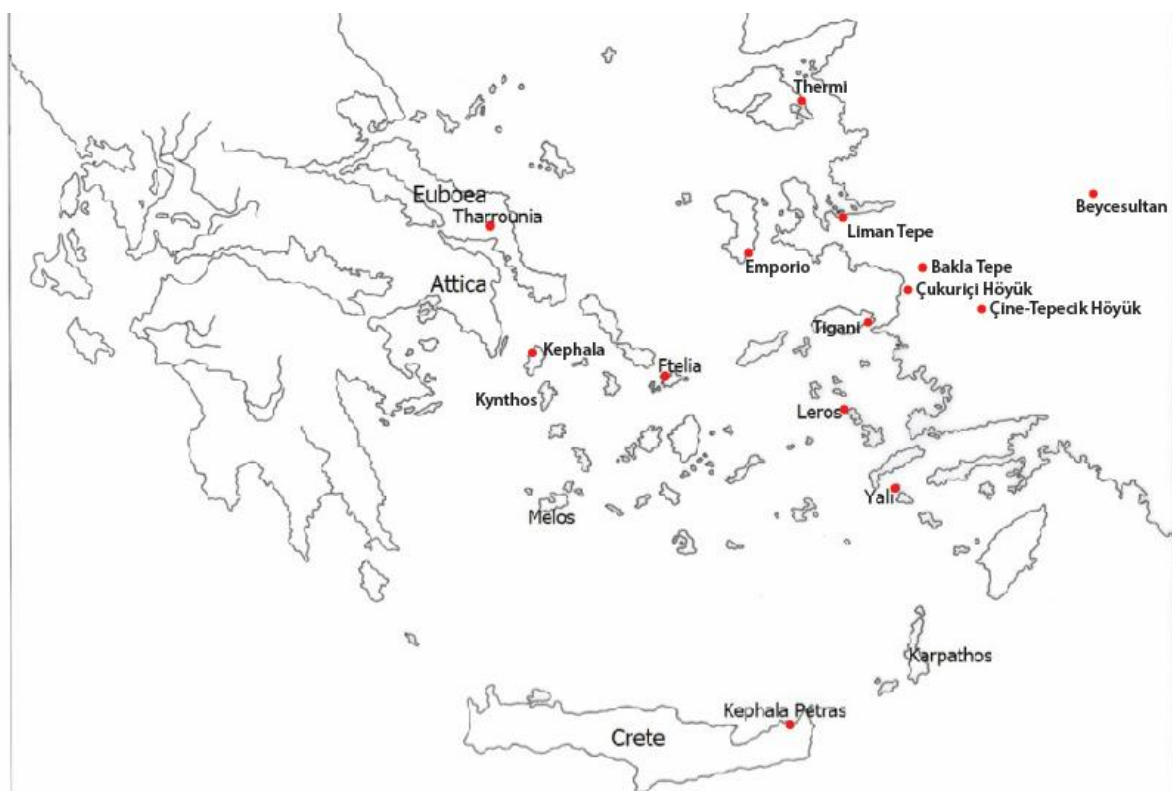


Figure 1: Yali in the Context of the Aegean, with some key sites noted.

APPROX. DATES B.C CALIBRATED	YALI SAMPSON (1985/2008)	ANATOLIAN COAST	GREEK MAINLAND	CYCLADES	CRETE TOMKINS (2007)	THESSALY	NORTHERN GREECE	S.BALKANS
c.45/4400 – c.3000	LAN4/LNI Ib YALI III	L.CH.4 BEYÇESULTAN XXIV-XX KURUÇAY 3 KUM TEPE IB EMPORIO VII/VI TIGANI III	FN DEMOULES & PERLÈS Phase 5 AYIA IRINI I	AYIA IRINI I	FN IV KNOSSOS (IC) KEPHALA PETRAS (NEO. BLG.) c.3300 - 3000	MICROTHIVES PETROMAGOUŁA OTZAKI B.C	KASTRI SITAROI III B- C DIKILI TASH IIA RACHMANI II	E. CHALCLOLITHIC BABANJ HURN IA KARANOVO IV KONDRADERMEN VINČA B KALOJANOVEC VINČA KARANOVO III (VESELINOVO)
		L.CH.3 BEYÇESULTAN XXVII – XXV KURUÇAY 6A KUM TEPE IIIB	KEPHALA (LATE) ATTIC -KEPHALA CULTURE	KEPHALA	FN III KNOSSOS II A c.3600 - 3300			
	LAN 3/LN IIa	FN II KNOSSOS II B c.3900 – 3600						
		L.CH. BEYÇESULTAN XL- XXXV KUM TEPE I B EMPORIO VII TIGANI II			FNIA c.4200 – 3900 KNOSSOS II – III			
					FRANCHTHI 5 THARROUNIA II KEPHALA (Early)	FN IA c.45/4400 – 44/4200 KNOSSOS II- III		

			ATHENIAN AGORA ATTIC KEPHALA		FN IB c.45/4400 – 44/4200			
c. 49/4800 – c. 45/4400	LAN2/LN 1b/ YALI II	EMBORIO VIII KUMTEPE IA BEÇIKTEPE KIZILBEL - LOWER BAGBAŞI TIGANI II - III	DEMOULES & PERLÈS Phase 4 GONIA CORYNCAN CAVE FRANCHTHI 5	ZAS, NAXOS BASE OF ZAS II B CAVE GROTTA SALIAGOS II – III THARROUNIA Ib	LN II KNOSSOS IV	DIMINI (CLASSIC) OTZAKI AGHIA SOPHIA	PARADIMI IV MAKRIYIALOS II DIKILI TASH II C DIKILI TASH II B SITAGROI III A SITAGROI III B-C DIKILI TASH IA-C	LATE NEOLITHIC VINČA MARICA I-IV KARANOV V

C.5300/ c.49/4800	LAN1/ LN Ia c.5000 YALI I	EMPORIO IX – X TIGANI I	LN DEMOULES LN & PERLÈS Phase 3 KITSOS ELATEIA FRANCHTHI FCP4	FTELIA THARROUNIA IA SALIAGOS I	LNI KNOSSOS V – VI	ARAPI TSANGLI - LARISSA	PARADIMI III SITAGROI II MAKRIYIALOS I DIKILI TASH I SITAGROI I PARADIMI I	KARANOVO III
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Figure 2: Chronological Relationship of Yali to 4th Millennium BC Communities in the Wider Aegean

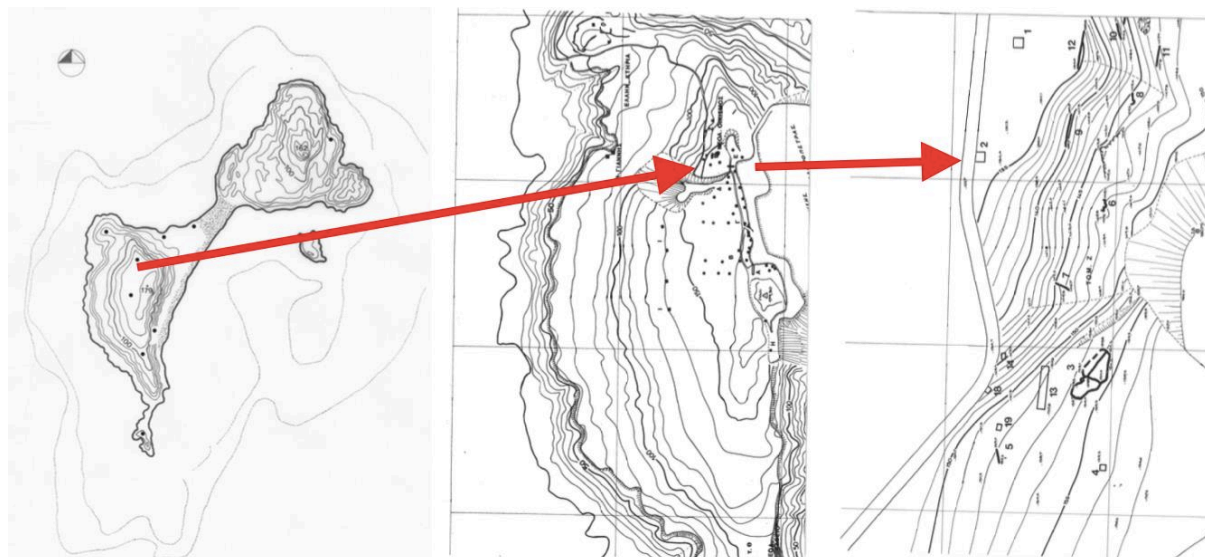


Figure 3: Spatial Context of Yali – the island › the higher plateau › the location of the main settlement with findspot of the crucibles in Z2.



Figure 4: The Apsidal Features in a building on Yali,
(After Sampson (1988, 54, 16a))

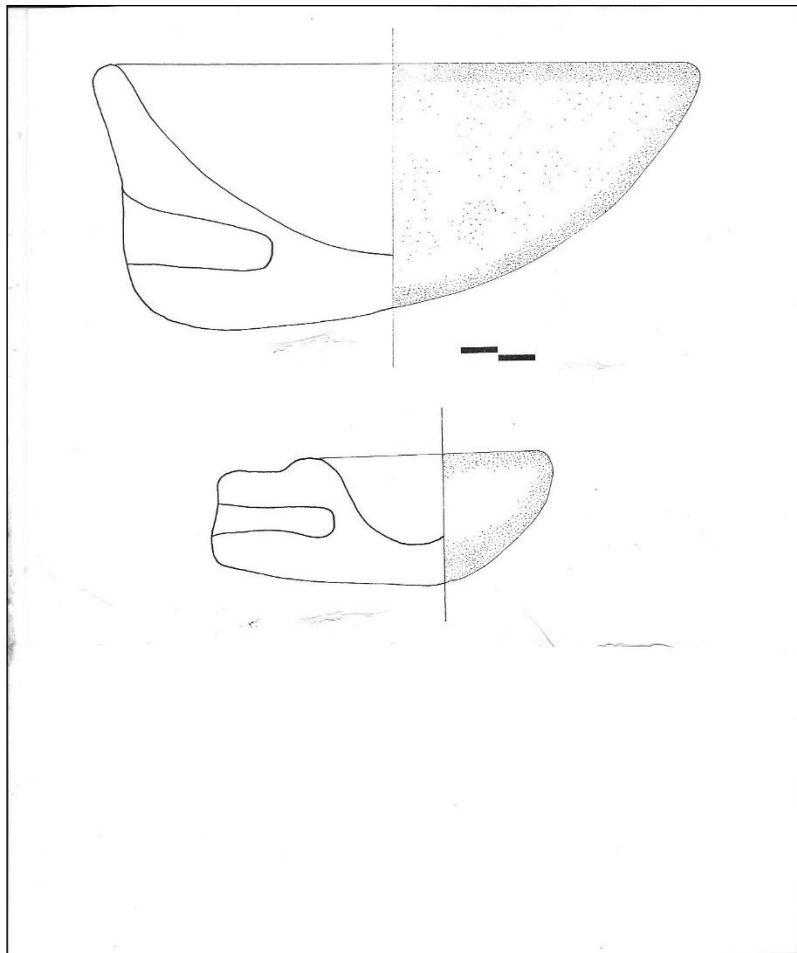


Figure 5. The Yali Crucibles

Element	Amount detected	Percentage Equivalent	
Copper	21.4	96.5	
Lead	0.014	0.063	
Tin	0.476	2.14	
Arsenic	0.285	1.28	
Iron	Trace		
Zinc*			
Nickel*			
Cobalt*			
* tested for but not found			

Figure 6. Chemical Composition of Copper in Crucible A (AAS),
(After Dr K. Assimenos, National Museum of Athens)

CRUCIBLE COPPER AND LEAD RIVET		Element		Concentration %
YALI 1		Pb	Lead rivet	100%
A F664 YALI 2		Cu		71.3
		Fe		?
		Mn		?
		As		?
		Ni		0.03
		Cd		0.02
		V		0.006
		Sb		?
		Zn		0.03
		Co		?
		Sn		0.002
B F645 YALI 3		Cu		95.6
		Fe		?
		Mn		?
		As		?
		Ni		0.09
		Cd		0.07
		V		0.02
		Sb		?
		Zn		0.02
		Co		?
		Sn		?

<u>Sample Name</u>	<u>Element</u>	<u>Intensity (cps)</u>	<u>Conc from OES(ppm)</u>	<u>Dil factor</u>	<u>Sample conc (ppm)</u>
F644 : YALI 2 A**				1498.1 3	
	Cu	4147769 7	475.9		712960.1
	Fe	357181.4			
	Mn	43171.4			
	As	9707.9			
	Ag	7843.9			
	Ni	4471.6	0.217		325.1
	Cd	1721	0.115		172.3
	V	1461.7	0.038		56.9
	Sb	554.2			
	Zn	553.1	0.214		320.6
	Co	41.2			
	Sn	32	0.079		118.4

F645 : YALI 3 B				10660. 98	
	Cu	7809869. 2	89.67		955970.1
	Fe	11978.6			
	Ag	3448.9			
	Mn	1502.1			
	As	446.7			
	Ni	391.5	0.084		895.5
	V	180.9	0.022		234.5
	Zn	94.3	0.205		2185.5
	Cd	75	0.066		703.6
	Sn	52.8	0.083		884.9
	Sb	39.8			

Figure 7: Chemical Composition of Copper in Crucibles A and B (Optimal Emission Spectroscopy Results of Two Copper Products from Yali Crucibles (normalised data (a) and raw data (b)), after Professor R. M. Ellam, Scottish Universities Research and Reactor Centre).

	$^{208}\text{Pb}/^{206}\text{Pb}$	$^{207}\text{Pb}/^{206}\text{Pb}$	$^{206}\text{Pb}/^{204}\text{Pb}$	No. Figs.
Crucible A-Large	2.06197	0.83196	18.864	1
Crucible B - Small	2.06438	0.83241	18.8616	2

Figure 8. Lead Isotope determinations of Copper from Crucibles A and B. (After Professor N. H. Gale, Laboratory for Archaeometry, University of Oxford, www.oxalid.ox.ac.uk)

	$^{208}\text{Pb}/^{206}\text{Pb}$	$^{207}\text{Pb}/^{206}\text{Pb}$	$^{206}\text{Pb}/^{204}\text{Pb}$	No. in Fig. 8,9
Crucible A - Cu	2.065928	0.833934	18.96532	3
Crucible B - Cu	2.067907	0.833972	18.99244	4
Rivet – Pb	2.063687	0.832466	18.99888	5

Figure 9: Lead Isotope Determinations of Copper from Crucibles A and B and Lead Rivet, (after Professor R. M. Ellam, Scottish Universities Research and Reactor Centre, University of Glasgow)

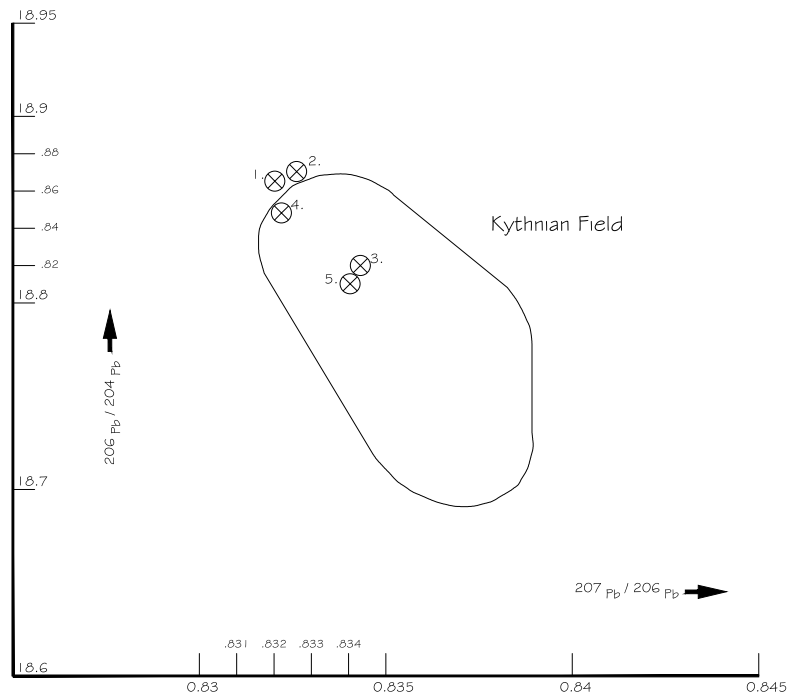


Figure 10: Lead Isotope Results (After Gale and Ellam. Samples 1 and 2 = Gale's analyses for Crucible A and B; Samples 3, 4 and 5 = Ellam's analyses for Crucible A and B and lead rivet, respectively, as they relate to the Kythnos field (determined by Gale and Stos Gale (1986).

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